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X-Ray Topography of Ruby Crystals

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ABSTRACT

X-ray topographs were made from nine ruby crystals submitted to NRL for examination. The crystalline planes examined were (0,0,12), (0,3,0), and (1,1,0). Most of the crystals showed growth imperfections in the form of striations perpendicular to the (1,1,0) planes.

PROBLEM STATUS

This is an interim report on one phase of the problem; work on other phases is continuing.

AUTHORIZATION

NRL Problem N01-09
ARPA Order 306-62

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X-RAY TOPOGRAPHY OF RUBY CRYSTALS

INTRODUCTION

Ruby crystals for lasers often show growth imperfections that are most easily recognized by x-ray topography, that is, an x-ray intensity map of the variations in orientation within a single crystal. The purpose of this report is to describe a method for making x-ray topographs and to show its apparent application to the examination of a number of ruby laser crystals.

Nine ruby crystals (Fig. 1) submitted to NRL have been examined by x-ray topography as a qualitative means of distinguishing gross crystalline imperfections. Four of the crystals were cut in the form of cubes, and it was possible to obtain diffraction topographs from three mutually perpendicular sets of crystalline planes, namely (0,0,12), (0,3,0), and (1,1,0). The other crystals were cut in various shapes, and sometimes only one set of planes was suitably oriented for topography.

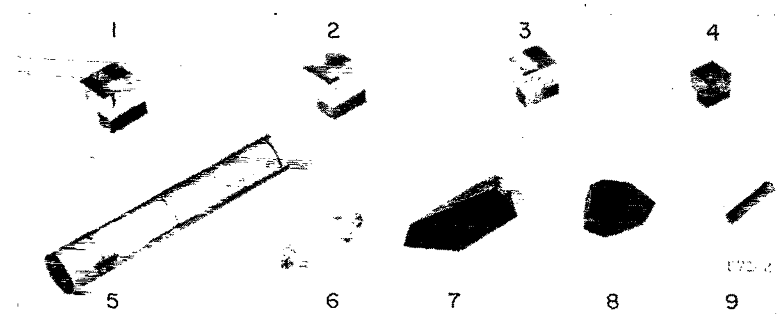

Fig. 1 - Ruby crystal specimens

X-RAY METHOD

The x-ray diffraction arrangement at NRL gives results similar to those of Schulz (1) or Berg and Barrett (2,3) but employs a modified geometry and is considerably faster. It is set up on a standard Philips diffractometer, Fig. 2, and shown schematically in Fig. 3. Characteristic CuKα radiation from the line focus of the x-ray tube diverges to the crystal. If the crystal planes were properly curved for the focusing circle, the incident x rays would be diffracted simultaneously from all portions of the crystal and would give the diffraction topograph, because each position on the film would correspond to a particular position on the crystal. Since, in practice, the crystal is flat, it must be rotated as shown by the arrow in order to approximate a curved crystal. Diffraction occurs sequentially from one end of the crystal to the other during rotation but strikes the same place on the film as it would from a curved crystal. The angular range of crystal rotation required depends on the length of the crystal. We have adopted a standard rotation of ± 3-degree θ, which is sufficient for crystals up to 2 inches in length. At a diffractometer speed of 1-degree θ per minute, the time required for a topograph is 6 minutes, as compared to several hours for some other methods of topography.

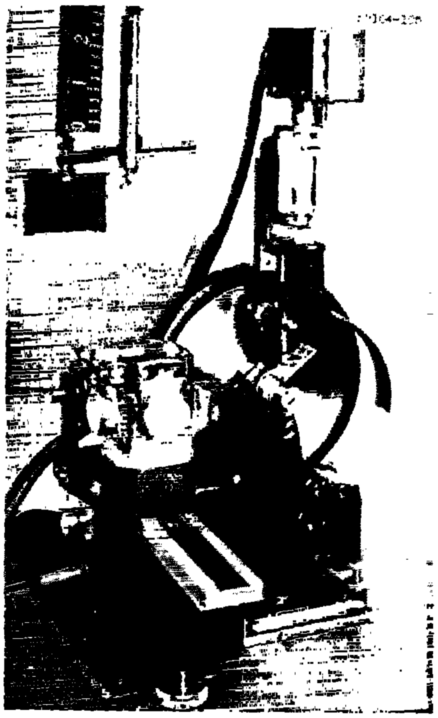

Fig. 2 - X-ray diffractometer modified to make x-ray topographs. A film cassette (insert) has been added.

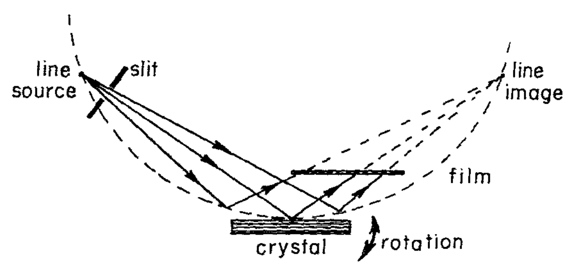

Fig. 3 - Schematic drawing of the x-ray geometry for topography

Ruby is a uniaxial crystal with a hexagonal crystal structure. Its optic axis is easily recognized between crossed polaroids. The important x-ray diffraction planes are (0,0,12), which is perpendicular to the optic axis, and (0,3,0) and (1,1,0) which are parallel to the optic axis. These three sets of planes are mutually perpendicular, and some of the crystals were cut in the form of cubes with these three planes as the cube faces. For such specimens it is possible to specify not only the diffracting plane but also the direction of the incident x-ray beam (this is important in determining the orientation of crystalline imperfections). Figure 4 shows how the cube faces are designated according to the markings on standard gambling dice; faces 1, 2, and 3 are always in the positions shown, and the numbers on opposite faces always sum to 7. For our purposes faces 1 and 6 are chosen perpendicular to the optic axis and parallel to the (0,0,12) diffracting planes. Faces 2 and 5 are parallel to the (1,1,0) diffracting planes, and faces 3 and 4 are parallel to the (0,3,0) diffracting planes. Topographs for the cube specimens are designated by three numbers: the number of the crystal, the number of the face closest to the source of x rays, and the number of the diffracting face. For example, in Fig. 4 let the crystal

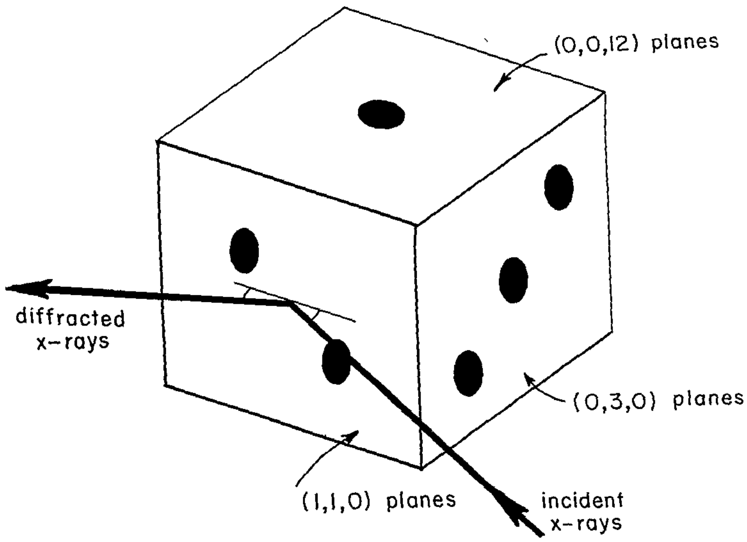

Fig. 4 - Designation of cube faces on ruby crystals according to the markings on gambling dice

be number 2; the face closest to the source of x rays is 3 and the diffracting face is 2; therefore the designation would be 2-3-2.

Some of the ruby crystals submitted were of odd shape, and only the plane perpendicular to the optic axis was recognizable. In such instances it is not easy to designate the incident direction, and only the diffracting plane can be specified.

RESULTS

Topographs of the nine specimens are shown in Figs. 5-7. Observations on each crystal are given below and are tabulated in Table 1. A complete designation for each specimen is given in the Appendix.

Crystal 1: Figure 5 shows a full set of 24 topographs (each of the six cube faces from each of four directions). The imperfection striations generally run parallel to the No. 3 cube face, that is, perpendicular to the (1,1,0) crystalline planes. The fact that the same imperfections do not appear in both 1-4-1 and 1-4-6 topographs of opposite faces means that the misoriented regions do not extend all the way through the crystal. The x-ray topographs show some degree of imperfection in each of the crystal planes, but comparative examination under crossed polarized light was not sensitive enough to show the imperfections except in the (0,0,12) planes.

Crystal 2: Figure 6 shows 12 topographs of crystal 2, which is much less perfect than crystal 1. The striations are more numerous and of greater contrast, indicating greater misorientation. Again the striations are seen in the (0,0,12) and (0,3,0) topographs and are perpendicular to the (1,1,0) planes. No topographs were obtained from the (1,1,0) planes.

Crystal 3: In Fig. 7, three topographs of crystal 3 are shown, one for each of the major sets of planes (0,0,12), (0,3,0), and (1,1,0). The degree of crystal perfection is similar to crystal 1.

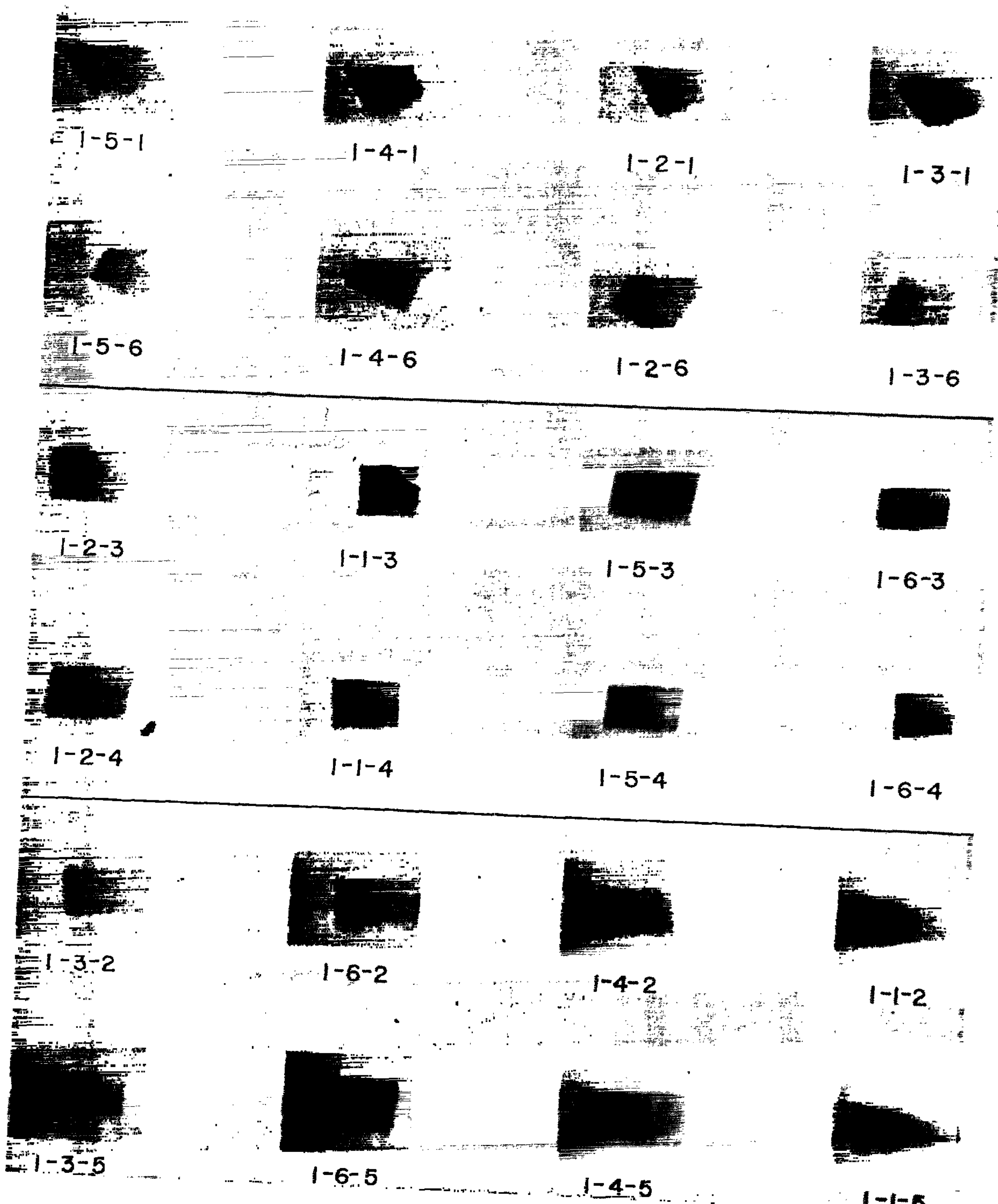

Fig. 5 - X-ray topographs of crystal No. 1

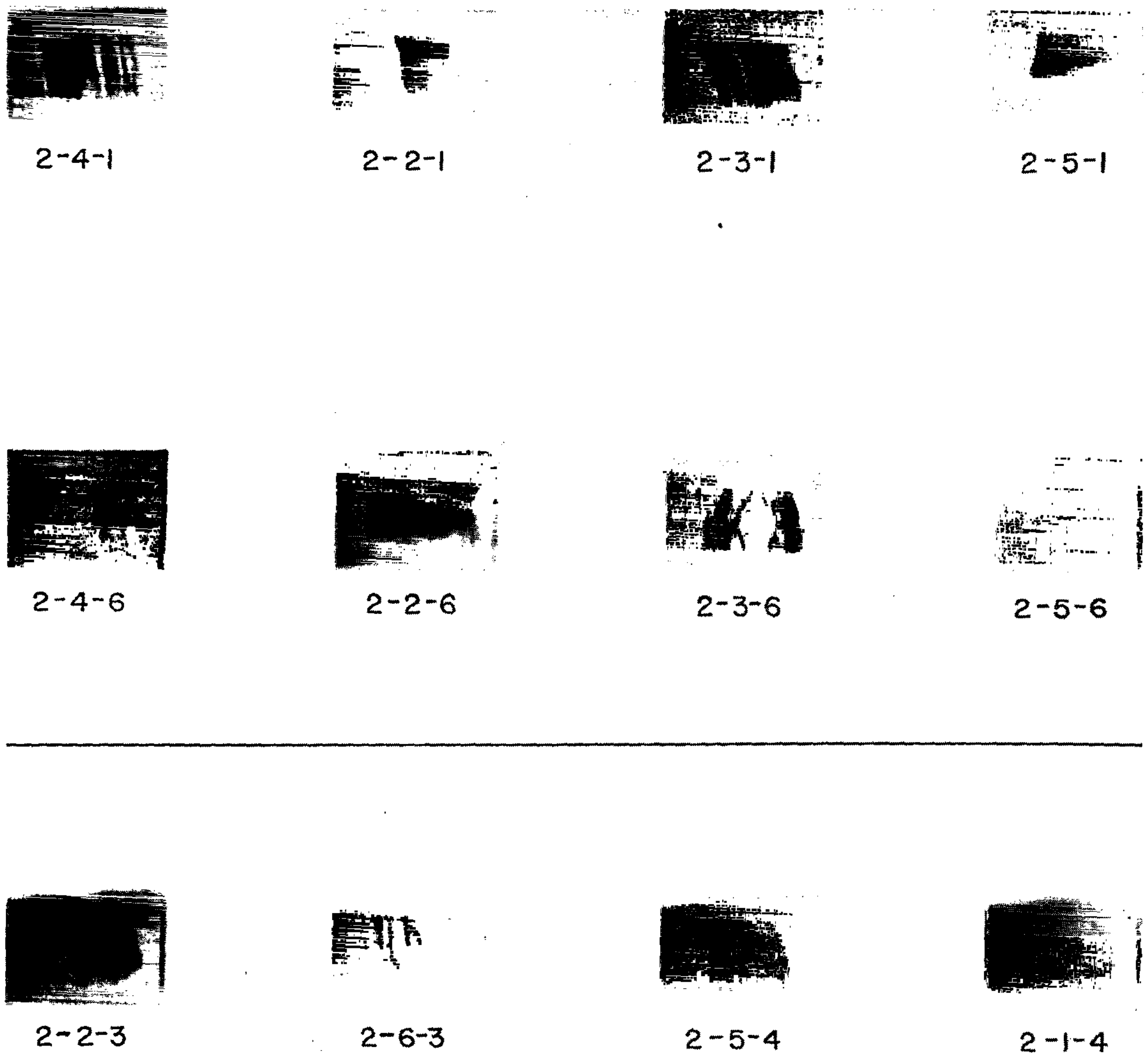

Fig. 6 - X-ray topographs of crystal No. 2

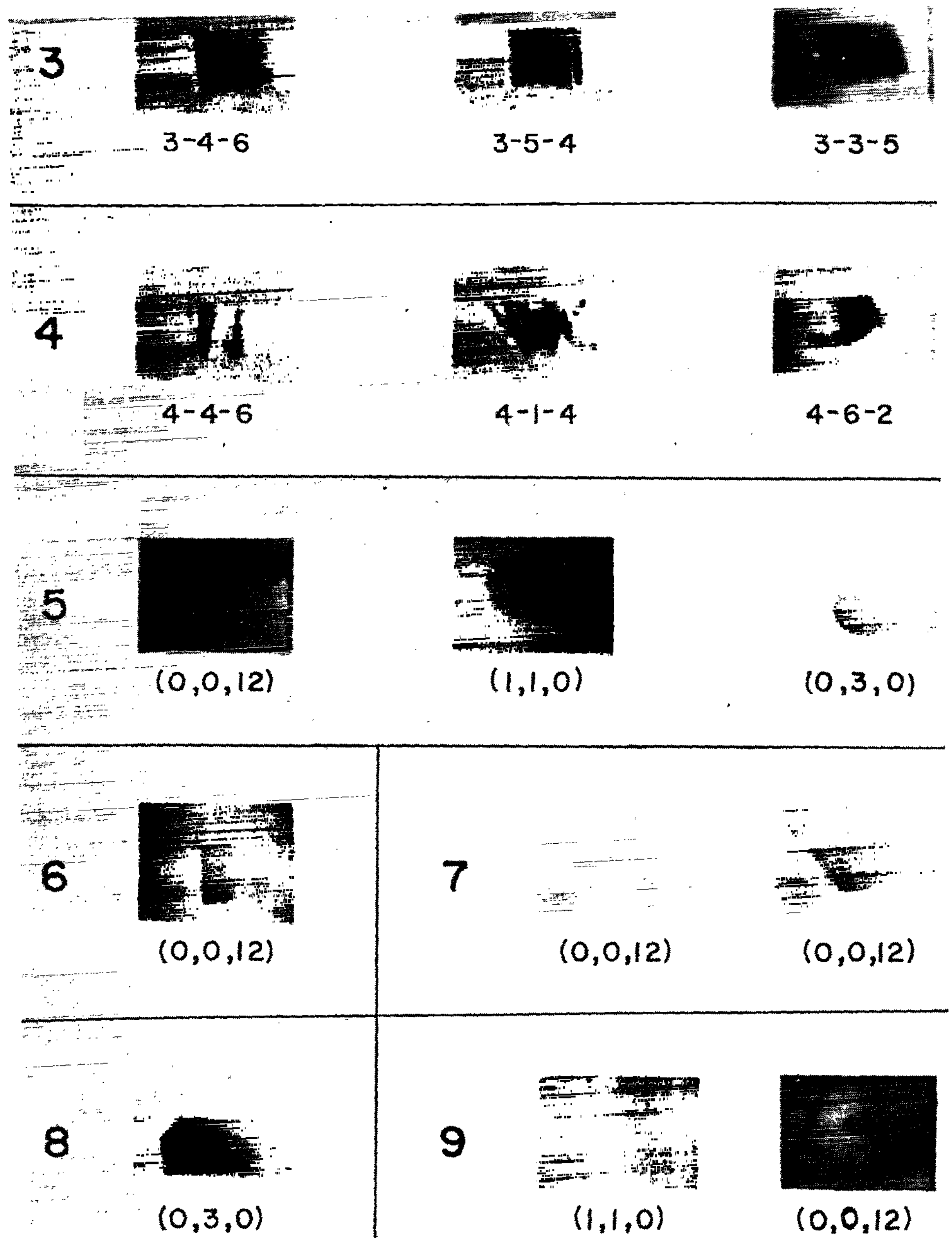

Fig. 7 - X-ray topographs of crystal No. 3 to 9. Note that crystals 5-9 are designated only by the crystallographic planes because of their irregular shape.

Table 1
Summary of Results

Crystal	Diffracting Planes	Observations
1	(0,0,12)	Few striations ⊥* to (1,1,0) planes Few striations ⊥ to (0,0,12) planes
	(0,3,0)	One defect ⊥ to (1,1,0) planes
	(1,1,0)	One striation ⊥ to (0,0,12) planes Several "scratches" of low diffraction intensity
2	(0,0,12)	Many striations ⊥ to (1,1,0) planes
	(0,3,0)	Many striations ⊥ to (1,1,0) planes
3	(0,0,12)	Few striations ⊥ to (1,1,0)
	(0,3,0)	Few striations ⊥ to (1,1,0)
	(1,1,0)	Few striations ⊥ to (0,0,12)
4	(0,0,12)	Many striations ⊥ to (1,1,0)
	(0,3,0)	Many striations ⊥ to (1,1,0)
	(1,1,0)	Poor diffraction Few striations ⊥ to (0,3,0)
5	(0,0,12) (0,3,0) (1,1,0)	Good diffraction with no striations
6	(0,0,12)	Good diffraction with no striations
7	(0,0,12)	Good diffraction but 1 or 2 small striations
8	(0,3,0)	A few striations
9	(0,0,12) (1,1,0)	Good diffraction with no striations

*Perpendicular

Crystal 4: Three topographs are shown in Fig. 7. As with crystal 3 all three major sets of planes are shown. There are many striations perpendicular to the (1,1,0) planes just as there were in Fig. 2.

Crystal 5: This crystal was cut in the form of a cylindrical rod (Fig. 1) with the optic axis perpendicular to the rod. In Fig. 7 the topographs are labeled according to the crystalline planes because the specimen shape precluded the designations used with the previous crystals. All of the topographs indicate a more perfect crystal than crystals 1-4. No striations or defects can be seen.

Crystal 6: Crystal 6 (Fig. 1) is a thin slab with the optic axis perpendicular to the large face. Only the (0,0,12) topographs could be obtained. As shown in Fig. 7, the perfection is good and is similar to crystal 5.

Crystal 7: The specimen submitted was of odd shape (Fig. 1). Only the (0,0,12) topographs were obtained, as shown in Fig. 7. One or two striations appear, and the perfection is less than that of crystals 5 or 6.

Crystal 8: This specimen was also of odd shape (Fig. 1). The only large face was approximately parallel to the (0,3,0) planes, as shown in the topograph of Fig. 7. A few striations are visible, and the overall perfection is similar to crystals 1 and 3.

Crystal 9: Crystal 9 was cut in the form of a small cylindrical rod (Fig. 1). As with crystal 5, the optic axis was perpendicular to the rod. Topographs of the (0,0,12) and (1,1,0) planes are shown in Fig. 7. No striations are visible and the perfection is similar to that of crystal 5.

CONCLUSIONS

1. In ruby crystals, x-ray topographs are a rapid and sensitive method for observing growth imperfections. It is easy to align the crystals for x-ray examination by finding the optic axis with cross-polarized light.

2. Of the crystals observed to date, the imperfections usually occur as striations perpendicular to the (1,1,0) crystalline planes. The significance of this with respect to growth processes is not known at this time.

3. A few of the crystals observed appear sufficiently free of imperfections to warrant a more sophisticated x-ray study, namely double-crystal spectrometer measurements of crystal mosaicness. Such measurements would require specimens in the form of cubes, such as specimen 1.

At present no definite relationships have been established between crystal imperfections and laser action, but it appears likely that the two are related. Intuitively, one might expect that more perfect crystals would be better lasers, but experimental correlation will be required for many crystals before final conclusions can be reached.

REFERENCES

1. Schulz, L.G., Trans. AIME 200:1082 (1954)

2. Berg, W., Z. Krist 89:286 (1934)

3. Barrett, C.S., Trans. AIME 161:15 (1945)

APPENDIX

DESIGNATION OF CRYSTALS

Report No.	Supplier Number	Supplier	Description
1	1830-16	Linde Co.	Unknown
2	T.S. 636	Thermal Syndicate, Ltd.	Flame fusion "vapor phase"
3	1739-18	Linde Co.	Unknown
4	T.S. 621	Thermal Syndicate, Ltd.	Flame fusion "vapor phase"
5	1963-19	Linde Co.	Unknown
6	1529-62	Linde Co.	Unknown
7	None	General Electric, Ltd.	Flux grown
8	None	General Electric, Ltd.	Flux grown
9	None	General Electric, Ltd.	Flux grown

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